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(54) Title: ANISOTROPIC FILM			
(57) Abstract Described are anisotropic films comprising a continuous elastic phase comprising a polyolefin elastomer; and a discontinuous phase oriented within the continuous phase such that the film exhibits anisotropic properties.			

ANISOTROPIC FILM

Field of the Invention

The invention relates to anisotropic films having a continuous phase and a
5 discontinuous phase.

Background

Elastic film materials, nonwovens, and other similar films have many
industrial and consumer uses. Among other uses, such materials are frequently
10 used, for example, in the disposable or personal use garment product area, garment
meaning a product used on or in association with a body (human or animal).
Specific such uses include disposable diapers, training pants, incontinence articles,
sanitary napkins, bandages, surgical drapes and gowns, medical nonwovens, face
masks, sport wraps and the like.

15 Generally, elastic films and materials can be formed from materials which
exhibit elastic properties in substantially all directions. However, for some
applications it is desirable to have materials which are primarily elastic in only a
single direction, i.e., materials that are anisotropically elastic. A large amount of
work, and a large number of patent applications and patents have been directed
20 toward providing such anisotropically elastic materials, with a wide variety of
solutions being provided.

A number of approaches have been successful in providing anisotropic
elastic films. One common approach has been to laminate an elastic web material to
a second web material that is easily stretched in one direction but not in the
25 transverse direction. To produce these "stretch-bonded laminates," an elastic film
or nonwoven materials, or a similar type of elastic web, is elongated in one
direction. While elongated, the elastic web is either continuously-bonded or point-
bonded to an inelastic web material. Afterwards, tension is released and the elastic
web is allowed to recover from its elongation. The attached inelastic web material
30 then puckers making the stretch-bonded laminate readily extensible in the direction
of the elastic web's elongation but not in the transverse direction. The laminate can

An aspect of the invention relates to anisotropic film having a continuous elastic phase comprising a polyolefin elastomer, and a discontinuous phase within the continuous phase. The film can be used by itself or in combination with other materials as a laminate material, e.g., for personal use garments.

5 As used herein, the term "anisotropic," refers to a film that exhibits elasticity and strength properties that are different when measured in one direction than in a second direction. The directions of measurement are conventionally referred to, e.g., as a "machine direction," "MD," or "inelastic direction," and a direction perpendicular to the machine direction, referred to as a "cross direction," "CD," or
10 "elastic direction." Elasticity and strength properties can be measured by a number of different physical properties of a film, including one or more of tensile strength, permanent set or set, and elastic force, etc. These properties are referred to herein as "film properties."

 The term "elastic" as used within the present description will be given
15 meaning as generally accepted in the elastic materials art, not inconsistent with the following: in terms of permanent set, an elastic material can be defined to recover at least about 80 percent of its stretched length after extension of 100% strain (double its initial length).

20 Detailed Disclosure

 The present invention relates to substantially anisotropic film comprising a continuous phase and a discontinuous phase.

 The continuous phase (also referred to herein as the "elastomeric phase") comprises an elastomeric polyolefin. This continuous elastomeric phase provides
25 the inventive film with elastic properties, and the elastomeric polyolefin can comprise any of a number of polyolefin materials that exhibit elastic behavior. The elastomeric polyolefin can have any degree of elasticity which, in combination with the discontinuous phase, provides a film having substantially anisotropic film properties as desired for a particular application. Preferably, in terms of permanent
30 set, a film of the elastomeric polyolefin can recover at least about 80 percent of its stretched length after extension of 100% strain (double its initial length), more

olefinic monomers or comonomers to produce a "homogeneously branched" ethylene polymers, as described, e.g., at pages 9 through 14 of PCT International Publication Number WO 95/33006 (International Application Number PCT/US95/06903). Therein it is described that elastomeric ethylene/alpha-olefin copolymers can be prepared by conventional polymerization processes using Ziegler-type catalysts (e.g., zirconium and vanadium catalysts) as well as metallocene catalyst systems.

It is further understood that elastomeric polyolefin polymers such as those described in United States Patent Number 5,472,775 can be a homopolymer or copolymer prepared from suitable unsaturated monomers or comonomers including, for example, ethylenically unsaturated monomers which can be straight chain (alkylenes such as ethylene, propylene, etc.) or cyclic (e.g., 2-norbornene), conjugated or non-conjugated dienes, polyenes, etc. Specific examples of suitable monomers and comonomers include C₂-C₂₀ alpha olefins such as ethylene, propylene, isobutylene, 1-butene, 1-hexene, 4-methyl-1-pentene, 1-heptene, 1-octene, 1-nonene, 1-decene, etc. For polyethylene elastomeric copolymers the density of the copolymeric material, and therefore its elastomeric properties, can be related to the amount of comonomer reacted with the ethylene monomer in that an increase in comonomer (e.g., a non-ethylene alpha-olefin) will generally decrease the density of the copolymeric material.

Examples of "metallocene" catalysts that can be used in the preparation of elastomeric polyolefins include those described in United States Patent Number 5,272,236 and PCT International Patent Application Number PCT/US96/14847 (International Publication Number WO 97/10300), those commercially available under the trade name INSITE™ catalysts from Dow DuPont, as well as other metallocene catalysts that are commercially available from other commercial sources.

Elastomeric polyolefins are also commercially available, for example: from Exxon Chemicals under the trade name of DEX Plastomers, e.g., EXACT 3000 and 4000 series plastomers, SLP-9000 and SLX-9000 series plastomers, and 2M004, 2M005, 2M007 series plastomers; from Mitsui Chemical Company under the trade

with the continuous phase (also for ease of processing). Preferred materials for use as the discontinuous phase include polystyrene, polyamide, polyesters such as polybutylene terephthalate (PBT), and mixtures thereof. Such materials are known in the chemical materials art, and are commercially available, for example, under the following trade names: Pocan 1300, 1600 from Bayer, PS 144c glasklar from BASF, PA12 from EMS (Grilamid L20G), PP7060S from Fina, LDPE Finathene LB520-0.

The film should contain a sufficient amount of the discontinuous phase to allow the discontinuous phase, when properly oriented, to cause the film to exhibit anisotropic behavior. The exact composition of the film and the amount of discontinuous phase relative to continuous phase can depend on numerous factors, including the desired strength and elasticity properties of the anisotropic elastic film, and the degree to which these properties vary anisotropically. It can be desired to consider the effect of film composition on more than one of the properties of strength and elasticity, and in more than one direction. That is, it might be important to find a balance of strength and elasticity in a combination of film directions (e.g., machine and cross directions). As an example, it may be important to consider that increasing the strength of a film in the machine direction can cause an increase of the elastic force and the permanent set in the cross direction due to a filling effect caused by replacement of the elastomeric polymer with the inelastic material of the inelastic phase.

When using the preferred extrusion techniques described below to prepare the anisotropic film, processing considerations may be the limiting factor defining the amount of discontinuous phase present in an anisotropic film. Specifically, when the materials of the continuous and discontinuous phases are extruded to form a film, the material of the discontinuous phase must generally comprise less than half of the film materials in order to be discontinuous. By extrusion methods, the use of about 45 percent of one phase can allow that material to constitute a fibrous discontinuous phase. And, although amounts outside of this range might also be desirable, preferred amounts of the discontinuous fiber phase within a given anisotropic film have been found to be in the range from about 1 to 40 parts by

microns. An anisotropic film of the invention having a discontinuous fibrous phase dispersed within a continuous phase will be referred to as "substantially anisotropic" if the film exhibits anisotropic properties that are enhanced versus a similarly-prepared homogeneous film comprised only of the continuous phase. The ratio of F10 in the machine direction versus F10 in the cross direction is preferably at least about 1.5, more preferably at least about 2, and even more preferably at least about 3.

The anisotropic film can be prepared from the materials of the continuous and discontinuous phases by any method that will provide a film having the discontinuous fibrous phase present within the continuous elastic phase in discontinuous domains properly shaped and oriented to provide a film having anisotropic properties. Generally, such a film can be produced by appropriately melting and casting the different components of the film to achieve such a result. Preferred methods of preparing the anisotropic film include extrusion methods, coextrusion methods, and blown film extrusion methods, all of which are well known in the art of producing films. In extrusion methods, the materials of the continuous and discontinuous film can be mixed together and melted by feeding them into one or more rotating screw extruders. The extruders then feed into a die or feedblock through which a die tip forms the extruded elastic film comprising a desired continuous elastic phase, with a desired discontinuous phase situated within the continuous phase as substantially parallel oriented fibers. As is well known, the extruded film can be cast upon a roller and the temperature reduced. Coextrusion methods and coextrusion/lamination methods, each of which are also well known, can be used to provide an anisotropic film laminate of the anisotropic film and one or more additional film layers laminated thereto. Also optionally, further processing such as additional stretching of the anisotropic film can be used if desired to further orient the film.

The anisotropic film material can be incorporated into multilayer products wherein the anisotropic film comprises an elastic layer in a multilayer film construction, such as can be understood from the disclosures of U.S. Patent Nos. 5,501,675, 5,462,708, 5,354,597, or 5,344,691. These references teach various

third, adhesive failure of the skin layer to the core layer under the microtextured folds with intermittent skin layer to core layer contact at the microtexture fold valleys. Generally, in the context of the present invention, all three forms of skin-to-core contact are acceptable. However, preferably the skin and core layers are in substantially continuous contact so as to minimize the possibility of delamination of the skin layer(s) from the elastic core layer.

Generally, the core layer to skin layer thickness ratio will be at least 3, preferably at least 5 but less than 100, and most preferably from 5 to 75.

The addition of the skin layer materials, as described in the above references, generally tends to further reinforce the anisotropic elastic film material layer in the machine direction. Also, following stretching and recovery in the cross direction (CD), the multi-layer film material exhibits substantially identical CD elastic properties to the elastic film core layer itself. As such, the CD stretched and recovered version of this multi-layer film exhibits enhanced anisotropic elastic behavior. However, prior to stretching and recovery the film generally is inelastic in both MD and CD directions.

The anisotropic elastic behavior in these coextruded laminates using the invention anisotropic film layer(s) can be accentuated as described in U.S. Patent No. 5,462,708 by subjecting a uniaxially stretched laminate to a deactivating heat treatment, while in the stretched condition. The heat treatment is such that the elastic recovery force of the elastic material is allowed to dissipate without substantially affecting the orientation of the inelastic skin materials. The heat treated laminate material is then stretched in a second cross direction and allowed to recover as described above. The resulting material is exceedingly strong in the original stretch direction and elastic in the cross direction. Machine direction orientation can also be used with other embodiments, with or without heat treatment, to provide additional anisotropic behavior to the invention anisotropic film material. This machine direction orientation can be up to the natural draw ratio of the fiber forming polyolefins of the inelastomeric polymer material portion. Generally this can be an orientation of up to six (6) times the original length of the film, although preferably from 2 to 5 times the original film length.

If the anisotropic film material is directly extrusion coated onto a nonwoven material, the nonwoven is generally contacted with the film less than about 2 seconds after the film has been extruded from the die tip so as to contact the nonwoven while it is still substantially in a heat softened state.

5 The invention anisotropic elastic film, whether a single layer film, a multi-layer film, or a laminate, can be used extensively in disposable or limited use garments and the like requiring an elastic that exhibits generally cross directional elasticity. For example, the material can be used extensively as an elastic in a disposable diaper such as waist band elastic, elastic side panels, or elastic ear
10 portions or in disposable training pants requiring specific zones of elasticity in order to create a tight-fitting, conformable garment. When used, the invention anisotropic elastic film material would generally be unwound from a roll and cut into suitable sizes and shapes for use in elasticating the disposable garment. The relatively inelastic behavior of the anisotropic film in the machine direction enables the film to
15 be more easily handled and cut into specific forms on conventional film handling machinery without undesirable elongation of the elastic (e.g., causing loss of film tension on the manufacturing line) in the machine direction. The invention material, when cut into appropriate shapes, can be applied in a conventional manner as is known in the art.

20

Test Methods

Measurement of F10 and F10 Ratio

Strips of elastomeric film measuring 2.54 cm by 15 cm by approximately 50-100 micron thick were cut along both the machine direction (MD) and cross
25 direction (CD) of an extruded film sheet. The force required to stretch the samples 10 percent (the F10 force) was measured using a standard tensile test configuration as described in ASTM D 882-95a. The value received was normalized by dividing it by the sample thickness and multiplying that result by 100.

The F10 ratio is the dimensionless number calculated by dividing the F10
30 force required for stretching the elastomeric film 10 percent of its original length in the machine direction divided by the F10 force in the cross direction.

F23 Polybutylene terphthalate, available as POCAN B-1300 from Bayer, Leverkusen, Germany.

F24 Polybutylene terphthalate, available as POCAN B-1501 from Bayer, Leverkusen, Germany.

5

Non Fiber forming, compatible materials

C25 Polypropylene, available as Finapro PPH 7060S from Fina Chemicals, Brussels, Belgium.

10 Additives/Other

A51 CaCO_3 masterbatch available as Omyalene G200 from OMYA GmbH, Koeln, Germany.

Example 1

15 An elastomeric film was prepared by extrusion using a single-screw extruder having a screw diameter of 45mm and a length/diameter ratio of 30 : 1, commercially available from Plastikmaschinenbau, Kehlberg, Germany. The barrel was heated in 5 zones to temperatures of 210, 220, 230, 235 and 240 °C, respectively, the temperature in the die had a temperature of 220°C.

20 Polyethylene pellets having a density of 0.863 g/cm³ (70 parts, available as ENGAGE 8180 from Dow DuPont Elastomers, denoted as B1), polystyrene pellets (25 parts, available as Polystyrol 144 CKG-2 from BASF, denoted F21), and CaCO_3 masterbatch available as Omylane G200 from OMYA were fed by gravity into the extruder. The extruder exit was fitted with a 400mm wide slot die with a
25 gap of 200μ.

The film was formed by casting onto a matte finish stainless steel roll, which was cooled to approximately 20°C with chilled water. The final film was wound into a roll at a speed of about 8 m/min and stored in roll form at approximately 22°C and had a thickness of about 100 microns.

30

Comparative Example 1

Table 1 Example 1 and comp.1 contain 5% CaCO₃ masterbatch A51

Example	PE type, parts	PE, density	Fiber-forming polymer type, parts	Perm. set (50%), % in c.d.	Perm. set (100%), % in c.d.	F10 ¹⁾ (MD) [N]	F10 ¹⁾ (CD) [N]	F10 ratio	Yield point, [N] in MD ¹⁾	Yield point, % elong in m.d..
Ex. 1	B1,	0.863	F21, 25	8	20	25.6	4.15	6.16	23.5	7.3
Comp. 1	B2	0.868	C25, 25	10	30	8.77	6.77	1.29	7	26
Comp. 2	B6	0.922	F21, 20	breaks at 12.1%	-	42.6	23.14	1.84	48.4	7.9

¹⁾ normalized to 100μ

Table 2 All examples contain 5% CaCO₃ masterbatch A51

Example	PE type, parts	PE, density	Fiber-forming polymer type, parts	Perm. set (50%), % in CD.	Perma. set (100%), % in CD	F10 ¹⁾ [N] (MD)	F10 ¹⁾ [N] (CD)	F10 ratio	Yield point in MD [N] ¹⁾	Yield point in MD, % elong.
2	B1	0.863	F21 0%	3	5	1.75	1.65	1.06	7.7	110
3	B1	0.863	F21 5%	4	10	6.4	2.0	3.2	7.8	43.3
4	B1	0.863	F21 10%	4	13	11.01	2.04	5.39	11.6	7.9
5	B1	0.863	F21 15%	6	15	14.88	2.9	5.13	15	7.9
6	B1	0.863	F21 20%	6	18	19.11	3.49	5.48	20.6	6.5
7	B1	0.863	F21 25%	8	20	21.77	4.15	5.25	23.5	7.3
8	B1	0.863	F21 30	13	30	27.05	7.57	3.57	29.6	7.1

¹⁾ normalized to 100μ

Table 3

Example	PE, type, parts, density	PE, type, parts, density	Overall density of the PE	Fiber-forming polymer, type	Fiber-forming polymer, parts
9	B1, 80, 0.863	-	0.863	F21	20
10	B1, 40, 0.863	B2, 40, 0.868	0.8655	F21	20
11	B2, 80, 0.868	-	0.868	F21	20
12	B2, 40, 0.868	B3, 40, 0.870	0.869	F21	20
13	B3, 80, 0.870	-	0.870	F21	20
14	B3, 40, 0.870	B4, 40, 0.885	0.8775	F21	20
15	B4, 80, 0.885	-	0.885	F21	20
16	B4, 40, 0.885	B5, 50, 0.908	0.8935	F21	20
17	B5, 80, 0.908	-	0.908	F21	20

Example 18

A film was prepared by the method described in Example 1 by mixing polyethylene having a density of 0.870 (80 parts, available as ENGAGE 8100 from Dow DuPont Elastomers SA CH-Geneva, denoted asB3) with a polyamide PA12 (20 parts, available as Grilamid L20 natur from EMS Chemie AG CH- Domat, denoted as F22).

Chemical composition of the film of Example 18 and its physical properties are summarized in Tables 5 and 6, respectively.

Example 19

A film was prepared by the method of Example 1 by mixing polyethylene having a density of 0.863 (80 parts, available as ENGAGE 8180 from Dow DuPont Elastomers SA CH, Geneva, denoted asB1) with polybutylene terephthalate (15 parts available as POCAN B1300 (TM) from Bayer, Leverkusen, Germany, denoted as F23) and CaCO₃ masterbatch (5 parts Omyalene G 200 from OMYA, Cologne, Germany denoted as A51).

Chemical composition of the film of Example 19 and its physical properties are summarized in Tables 5 and 6, respectively.

Example 20

A film was prepared by the method of Example 1 by mixing polyethylene having a density of 0.863 (80 parts, available as ENGAGE 8180 from Dow DuPont Elastomers SA CH, Geneva, denoted asB1) with polybutylene terphthalate (15 parts available as POCAN B1501 (TM) from Bayer, Leverkusen, Germany, denoted as F24) and the CaCO₃ masterbatch (5 parts Omyalene G 200 from OMYA, Cologne, Germany denoted as A51).

Chemical composition of the film of Example 20 and its physical properties are summarized in Tables 5 and 6, respectively.

Example 21

Polyethylene pellets having a density of 0.868 g/cm^3 (70 parts, available as ENGAGE 8150 from Dow DuPont Elastomers, denoted as B2) and polystyrene
5 pellets (25 parts, available as Polystyrol 144 CKG-2 from BASF, denoted as F21) and the CaCO_3 masterbatch available as Omylane G200 from OMYA were fed by gravity into the extruder.

Example 21 was repeated with the exception that the extruded film was oriented in the length direction by the following process. The film was first
10 preheated on a rolls with 65° and then the softened film was stretched between two nips, where the second nip was running at a higher speed than the first nip. The film was stretched from 115 micron to 86 microns and then allowed to cool. The film was then wound up upon itself and stored at approx. 22°C :

The properties of the oriented film were measured and compared to the un-
15 oriented film of the same material. The results show that orientation increased the strength of the film in the machine direction (MD). This is expressed by the force at yield normalized to 100μ .

We claim:

1. An anisotropic film comprising:
a continuous elastic phase comprising a polyolefin elastomer; and
5 a discontinuous phase oriented within the continuous phase such that the film exhibits anisotropic properties.
2. The film of claim 1, wherein the polyolefin elastomer comprises polyethylene.
- 10 3. The film of claim 2, wherein the polyolefin elastomer comprises a polymer derived from monomeric units consisting of polyethylene and one or more comonomer.
- 15 4. The film of claim 3, wherein the comonomer comprises a C₂-C₂₀ alpha olefin.
5. The film of claim 4, wherein the alpha olefin is chosen from the group consisting of propylene, isobutylene, 1-butene, 1-hexene, 4-methyl-1-pentene, 1-
20 heptene, 1-octene, 1-nonene, 1-decene, 2-norborene, and mixtures thereof.
6. The film of claim 1, wherein the polyolefin elastomer has a fifty percent permanent set value of less than about 35 percent.
- 25 7. The film of claim 6, wherein the polyolefin elastomer has a permanent set of less than about 25 percent.
8. The film of claim 1, wherein the polyolefin elastomer has a density below about 0.92 grams per cubic centimeter.

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19. The film of claim 1, wherein the film has an F10 ratio of at least about 1.5.
20. An anisotropic elastic film laminate comprising:
an anisotropic layer comprising:
5 a continuous elastic phase comprising a polyolefin elastomer;
and
a discontinuous phase oriented within the continuous phase;
and
a laminate layer laminated to the anisotropic layer.
- 10 21. A personal use garment comprising an anisotropic film, the anisotropic film comprising a continuous elastic phase comprising a polyolefin elastomer, and a discontinuous phase oriented within the continuous phase.

INTERNATIONAL SEARCH REPORT

Int. ional Application No

PCT/US 98/17312

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C08J5/18 B32B27/32 //C08L23:08,C08L25:04,C08L67:00,
C08L77:00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C08J B32B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 93 16863 A (EXXON CHEMICAL PATENTS INC) 2 September 1993 see the whole document ---	1-21
Y	WO 95 05418 A (DOW CHEMICAL CO) 23 February 1995 see the whole document & US 5 472 775 A cited in the application ---	1-21
Y	WO 93 08221 A (DOW CHEMICAL CO) 29 April 1993 see the whole document & US 5 272 236 A cited in the application ---	1-21
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Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents :

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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